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Liquid Tin Anode SOFC JP-8 Start-up

ABSTRACT

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Liquid Tin Anode SOFC JP-8 Start-up

Prepared By

**CellTech Power, LLC,
131 Flanders Road, MA, 01581**

October, 2008

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Introduction:

The program effort began on the 1st of May 2008 as stated in contract W911NF-08-1-0115. This program follows programs #W911QY-04-2-003 and #W911NF-07-C-0032 that both demonstrated the unique fuel flexibility of CellTech Power's technology. In particular both of the aforementioned programs demonstrated the ability to run on JP-8 fuel without reforming or fuel processing. However the previous testing set ups involved additional reducing gases such as hydrogen and 95%Ar/5%H₂ during start up and cool down to protect the liquid anode tin and certain non-precious metal current collectors. The work done on this program utilized CellTech Power's Gen3.1 Liquid Tin Anode – Solid Oxide Fuel Cells (LTA-SOFC) running on JP-8 fuel (sulfur 1,400 ppm) only during startup and cool down. The use of JP-8 only during start up and cool down is particularly important since in the field there likely are no other reducing gases available.

The key results of these previous programs on JP-8 fuel using Gen 3.1 cells were:

- Direct JP-8 conversion to electricity without reforming & sulfur removal
- Peak power density of 120 mW.cm⁻²
- Peak efficiency of 40-45%
- 100 hr of operation without any significant performance degradation
- 4x reduction of the weight and volume compared to Gen3.0 cells (2005-2006)

In addition to the DARPA/ARL programs focused on JP-8 as a fuel, CellTech Power has also demonstrated operation on other carbonaceous fuels including bio-mass and coal using the new Gen3.1 single cell design. The unique fuel flexibility of LTA-SOFC technology is due to two reasons:

1. The liquid tin anode surface is not prone to sooting, which ensures a reaction site for the fuel to reduce tin oxide generated from the electrochemical reaction.
2. Its ability to utilize sulfur as fuel.

The reaction of liquid tin with various carbonaceous fuels has been described elsewhere [1-6]. However, the internal gasification reaction between the exhaust stream and solid carbonaceous fuels is of particular interest. The key chemical heterogeneous equilibrium for carbonaceous fuels is:



Where: CO_{2(g)} is the exhaust gas from the tin oxide reduction reaction

C_(s) is the carbon formed during JP-8 and other carbonaceous fuel thermal decomposition at operational temperature (1000°C).

$\text{CO}_{(\text{g})}$ is the product of the reaction that is a useable fuel for further tin oxide reduction.

Analysis of this equilibrium using HCS Chemistry Ver 4.1 software allows the spontaneity of the reaction to be plotted as a function of temperature. This trend, Figure 1, shows that the reaction is spontaneous above 700°C with a ΔG of -52.23 kJ , for equation 1, at 1000°C . Thus thermodynamics of the fuel gasification reaction dictate operation at temperatures above 700°C .

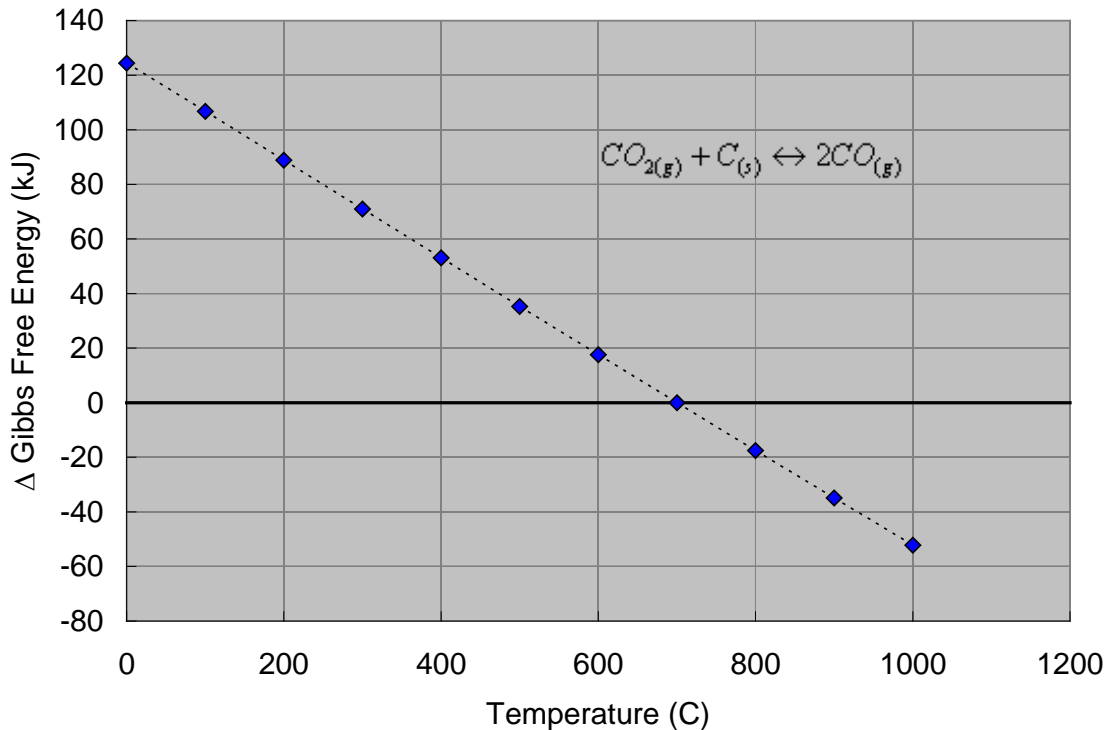


Figure 1: At 1000°C the reaction of solid carbon with carbon dioxide to form carbon monoxide is spontaneous as represented by the negative change in Gibbs free energy for the reaction.

A limiting factor to high fuel utilization is the solubility limit of oxygen in liquid tin. Work by CellTech Power has shown experimentally and theoretically that 80% fuel utilization is the practical limit to avoid detrimental precipitation of tin oxide at an operational temperature of 1000°C when hydrogen is used as a fuel. Nevertheless, similar to traditional SOFCs, 80% fuel utilization is normal and commercially viable. It is also suggested that the intended application, portable vs. stationary, is the most influencing factor for fuel utilization. In work CellTech Power has done for the Electric Power Research Institute (EPRI) and the Department of Energy on megawatt stationary systems there has been several system level designs for recycled anode exhaust, which would effectively achieve 100% fuel utilization. The design concept of recycled anode exhaust is common to large stationary fuel cell power plants. In contrast a portable system is unlikely to run at fuel utilization greater than 80% since the unused 20% fuel can be combusted to keep the fuel cell at the desired operation temperature. Minimization of

weight and size dictate portable design. As such it is typically beneficial to burn more energy dense fuel then it is to add bulk and weight to the design with addition insulation. The aforementioned discussion of the carbon gasification reaction and it relationship to fuel utilization illustrates the flexibility and uniqueness of CellTech Power's LTA-SOFC technology. CellTech Power's development of this technology for military application is focused on a squad and or man portable units.

In a practical cell designed for portability, the liquid tin has to be encased around the electrolyte to ensure orientation independence and robustness. CellTech Power has pioneered the development of a highly porous, >60%, structural ceramic material that separates the solid fuel from the tin and also holds the tin in intimate contact with the electrolyte. This part is referred to as the porous separator and the Gen3.1 iteration is illustrated in Figure 2. The upper limit of the pore size for this material is 200 μm for a head pressure less than 10 cm tin height. This limit is set by the intrinsic material property of surface tension for tin in a reducing atmosphere. A pore size greater then 200 μm will result in tin leaking through the porous separator. In addition to the pore size limitation of the porous separator, the cross sectional area has to be sufficient to ensure that the part can act as a load bearing structure. Since the porous separator part is tubular to follow the contours of the electrolyte the cross sectional area can be simplified to a single dimension limitation of wall thickness. Theoretically, the mass diffusion limitation for diffusion of carbon dioxide from the tin to the carbon and carbon monoxide diffusion back to the tin, will be inversely proportional to the pore size and directly proportional to the wall thickness of the porous separator. It is also clear that the tortuosity of the porous separator will also be proportional to mass diffusion limitation.



Figure 2: The Gen3.1 iteration of the porous separator is shown which was fabricated in house to a porosity of great then 60%

The challenge is to engineer a functional LTA-SOFC that will run off logistic military fuel for portable applications that maintains the tin in intimate contact with the electrolyte while minimizing the mass diffusion limitation caused by the porous separator. This was achieved on the DARPA/ARO # W911NF-07-C-0032 program with CellTech Power's Gen3.1 cell. A radical redesign of the Gen3.0 cell allowed 4 times the power to be achieved at a quarter of the weight and volume. The performance difference between the Gen3.1 and Gen3.0 cell is illustrated in Figure 3. However, initial heat up, cool down and benchmarking was done using hydrogen. Therefore a program that looked at start-up and function on JP-8 fuel as the only fuel was a logical next step.

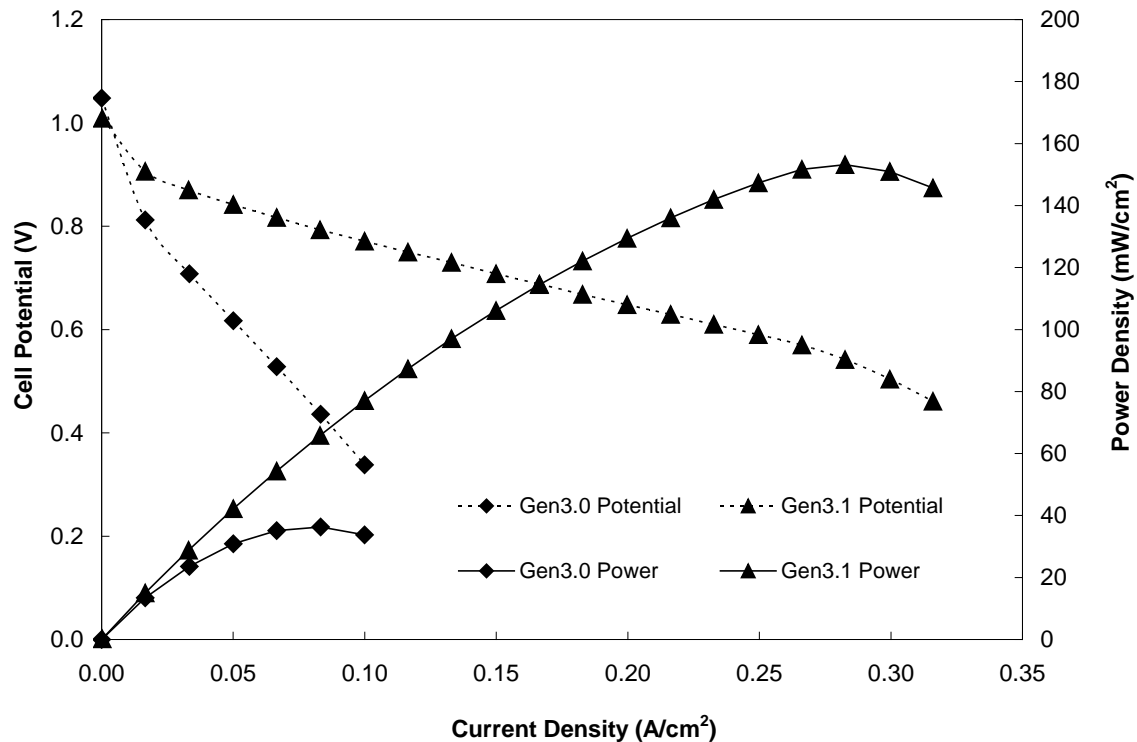


Figure 3: The Gen 3.1 cells have significantly less polarization compared to the Gen 3.0 cells, using JP-8 or hydrogen as a fuel, hydrogen-air performance plotted.

The current program fills the need to test startup and shutdown operation of the Gen3.1 design on JP-8 fuel. Of particular relevance was the materials interaction of the JP-8 with the porous separator and current collectors.

Deliverables:

Demonstration of feasibility of JP-8 only start up and cool down was the goal of this program. To achieve the goal, action items included:

1. Production of 5 LTA-SOFC cells for testing
2. List of materials and components which are areas of concern during start/stop.
List of potential solutions
3. Cell performance evaluation during testing including start up and cool down.
4. Development of a recommended procedure for JP-8 startup and shut down.

Technical Approach:

A reducing/inert atmosphere is required around the liquid tin anode during cycling between 400°C and operational temperature, 1000°C. This prevents detrimental formation of tin dioxide (SnO_2) as opposed to the formation of tin monoxide (SnO) which is reduced back to tin by the fuel without degrading any of the components. Also there

are non-precious metal current collectors at cell, stack and system levels that need to be protected from oxidation during startup and cool down. At operational temperature fuel (JP-8) is metered into the anode to reduce the tin monoxide (SnO) from the electrochemical reaction back to tin. A practical system will require JP-8 to provide a reducing atmosphere at the anode during thermal cycling. Thermal decomposition of JP-8 at elevated temperatures ($>100^\circ\text{C}$) will give a chemically reducing environment from the volatiles and from the soot reacting with oxygen to form carbon monoxide or carbon dioxide. However, this reducing atmosphere has to be managed to ensure all components susceptible to oxidation at elevated temperatures are protected. In addition the metering of the fuel into the anode chamber has to be controlled so sooting is not detrimental to the operation of the device. This program identifies and solves the aforementioned issues.

Results: – Materials and Component List

The Gen3.1 design is built around a centered cathode current collector with 10 mm diameter electrolyte tube. The tin is held against the outer surface of the electrolyte using a porous ceramic monolith. The porous ceramic has macro porosity of 60-65% to ensure unimpeded fuel flow to the liquid tin. The surface tension of the liquid tin anode prevents the liquid tin from penetrating the porous ceramic monolith. The test article used is illustrated in Figure 4. Materials and components which need to be considered during thermally cycling are listed below:

- Porous Separator
- Metallic interconnects that join the anode and cathode current collectors (hot zone – 1000°C) to the load cables (cold zone – ambient).
 - Metallic interconnection inside the anode chamber
 - Metallic interconnection outside the anode chamber
 - Soot build up and bridging between the anode and cathode metallic interconnects inside the anode chamber
- Fuel inlet port
- Anode chamber exhaust port

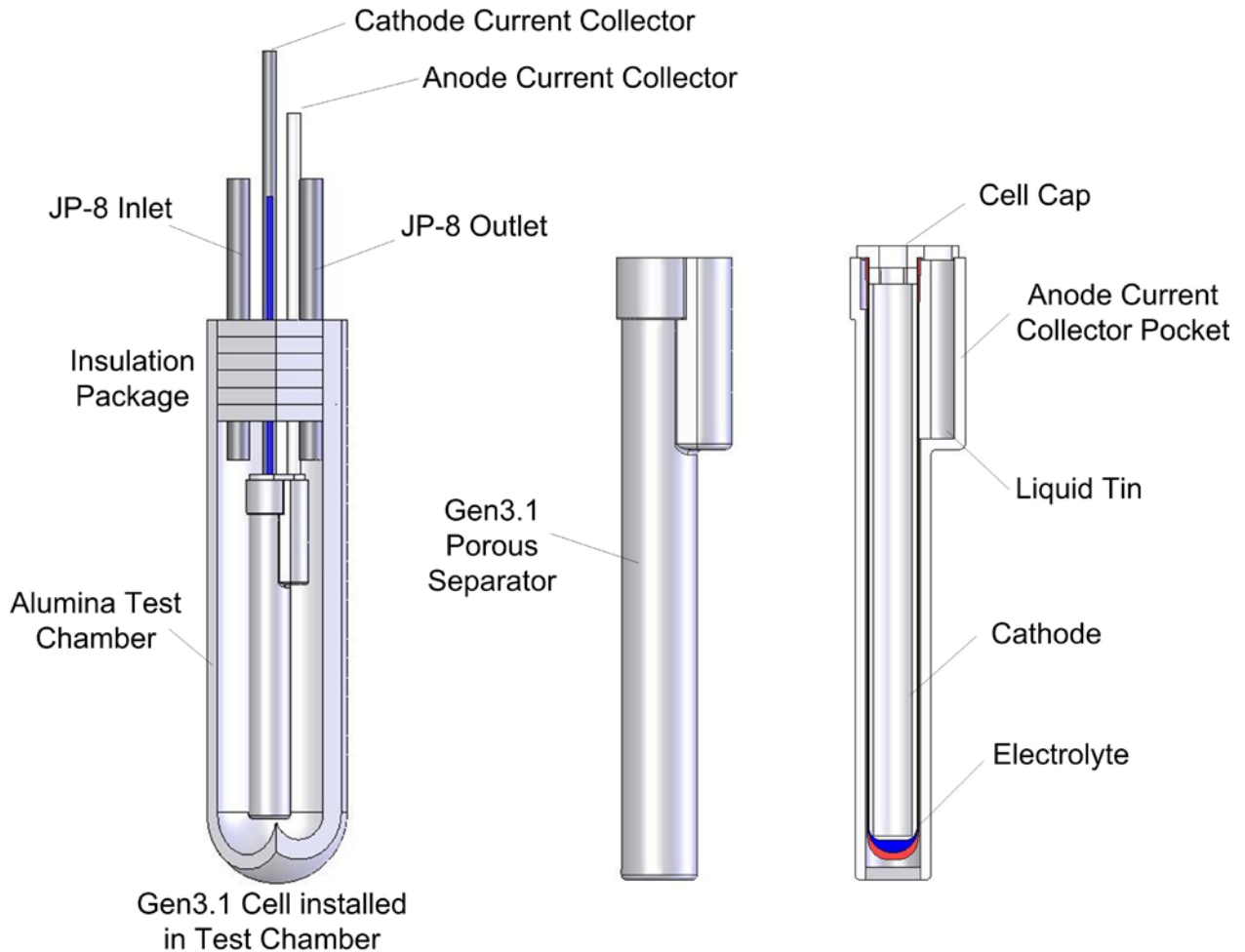


Figure 4: The Gen3.1 test article set-up showing areas of concern during heat-up and cool down on JP-8.

Result & Discussion: – Cell Performance

Several cells were thermally cycled on JP-8. As shown in Figure 5 & Figure 6 the cells showed reasonable performance on multiple cycles when subjected to very high JP-8 flow rates. Both cells were subject to flow rates of 50 $\mu\text{l}/\text{min}$ during cycling from 400-1000°C. This high fuel flow produced a dense layer of carbon as shown in Figure 7. This type of carbon formation was not witnessed when running cells at 1000°C steady state on JP-8. However, the porosity of the porous separator was still maintained, Figure 8. The dense layer of carbon was prevalent at the bottom of the cells on the outside of the porous separator. This layer significantly reduced the performance of the cells, Figure 9. However, the JP-8 flow rate was extremely high and is not a realistic value per cell at the system level.

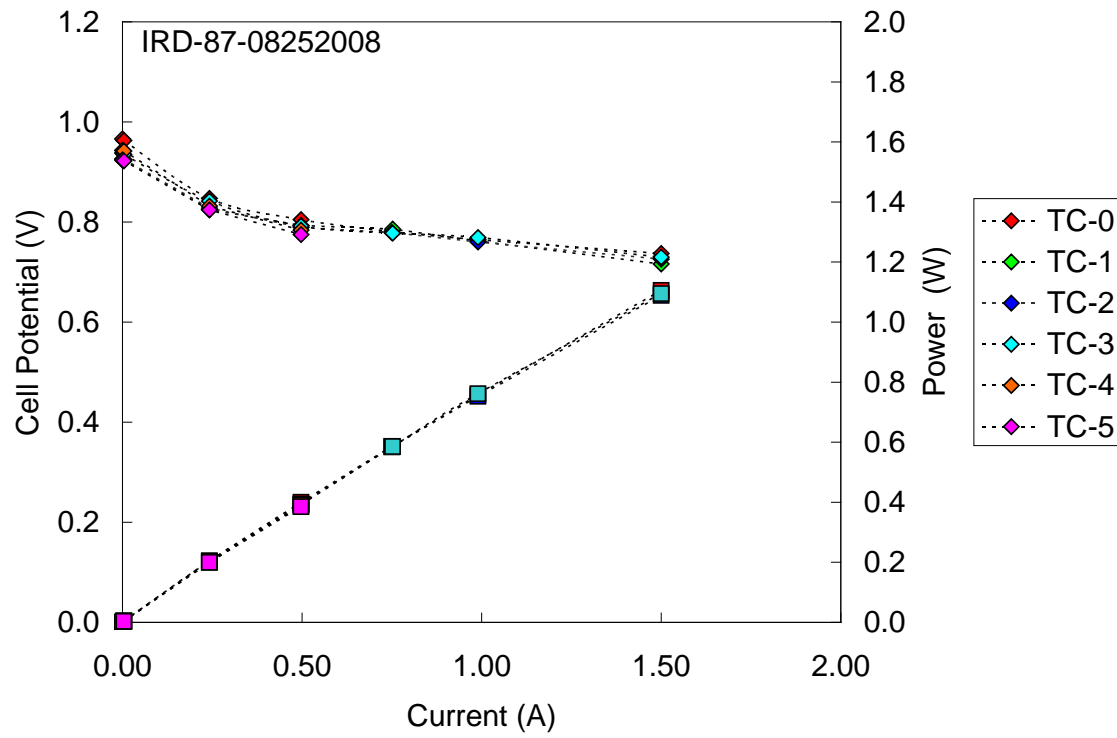


Figure 5: Five thermal cycles on JP-8 at a flow rate of 50 $\mu\text{l}/\text{min}$, show a reduction in maximum current due to sooting of the porous separator.

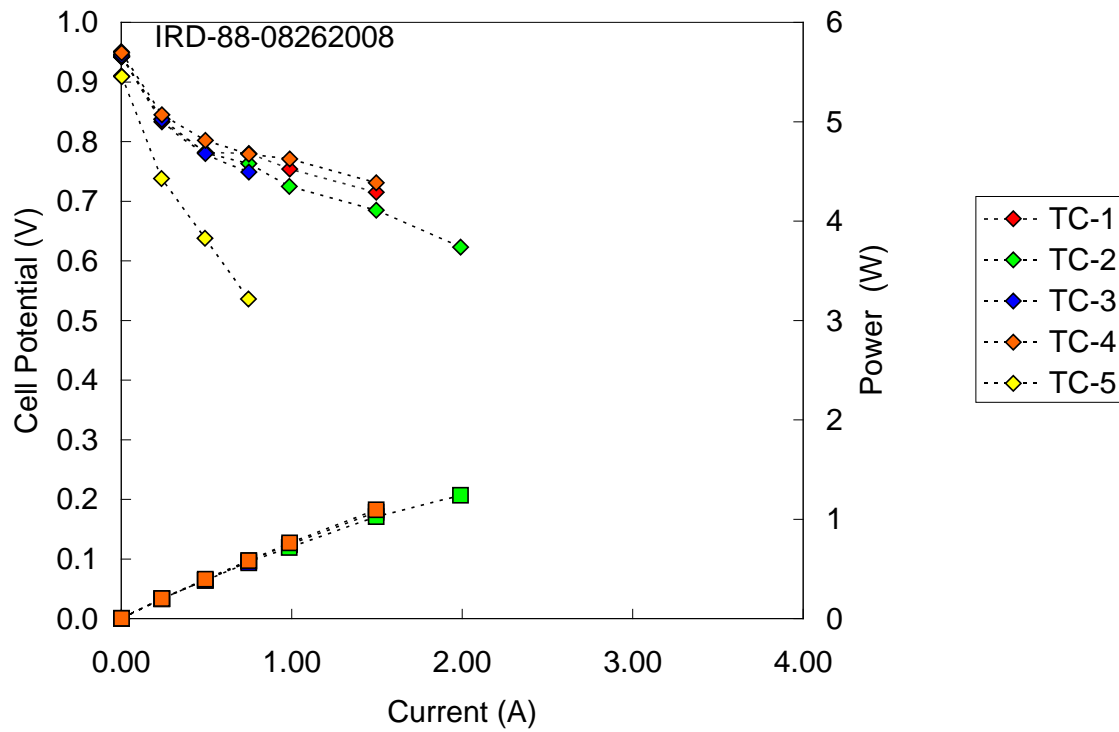


Figure 6: Five thermal cycles on JP-8 at a flow rate of 50 $\mu\text{l}/\text{min}$, show a reduction performance on the fifth cycle due to a testing issue.



Figure 7: Carbon build up on the bottom of the cell shut off the porous separator

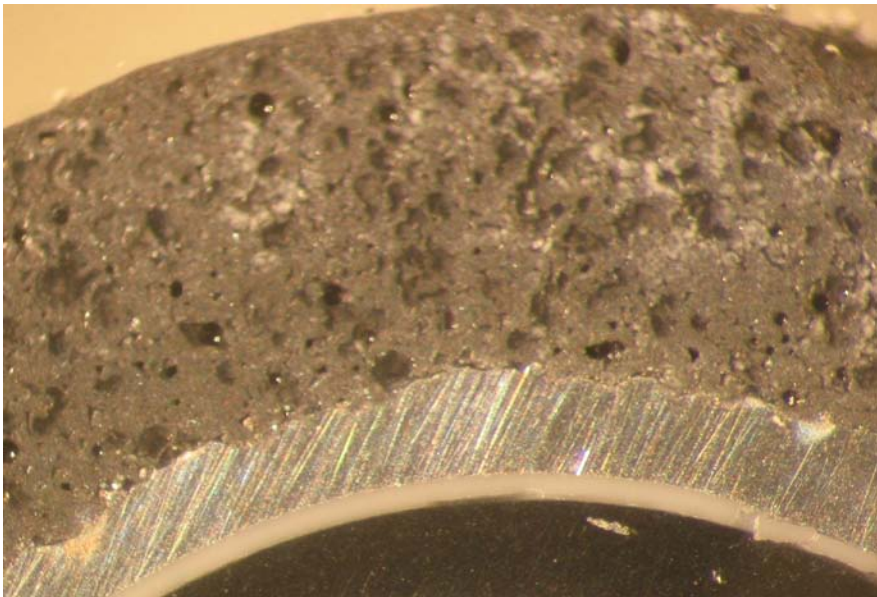


Figure 8: porous separator at the top of the cell showing no tin penetration and no significant sooting of the porous separator.

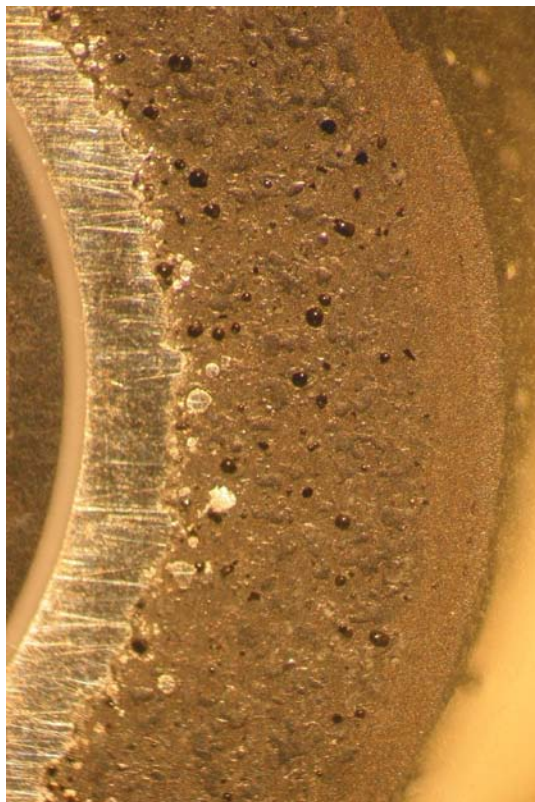
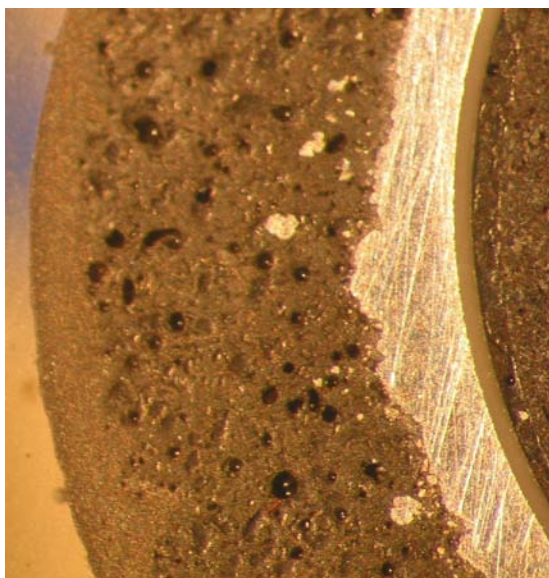


Figure 9: Porous separator at the bottom of the cell after 5 thermal cycles showing that the porosity though the separator is preserved, but a solid layer of carbon has built up on the out side of the porous separator.

Result & Discussion: – Potential Solutions

Porous Separator: The porous separator encapsulates the liquid tin around the electrolyte and provides the flow path for gas transport to and from the tin. Soot formation in the pores of the porous separator has not been shown to prevent electrochemical performance when deposited at 1000°C. However, the mass diffusion limitation is always a concern and it is suggested that the carbon forms a more dense layer at lower temperatures such as during start up or cool down. In previous work on JP-8 carbon build up at 1000°C has been highly porous and has apparently not prevented gas flow to and from the tin although limitation of soot build up on the porous separator is considered advantageous. An ideal scenario after a thermal cycle would be a porous separator that was lightly coated with amorphous carbon, without a visible build up of excessive soot. This was achieved with on a recent single cell test that exclusively used JP-8 as a fuel during thermal cycling and operation, Figure 10.

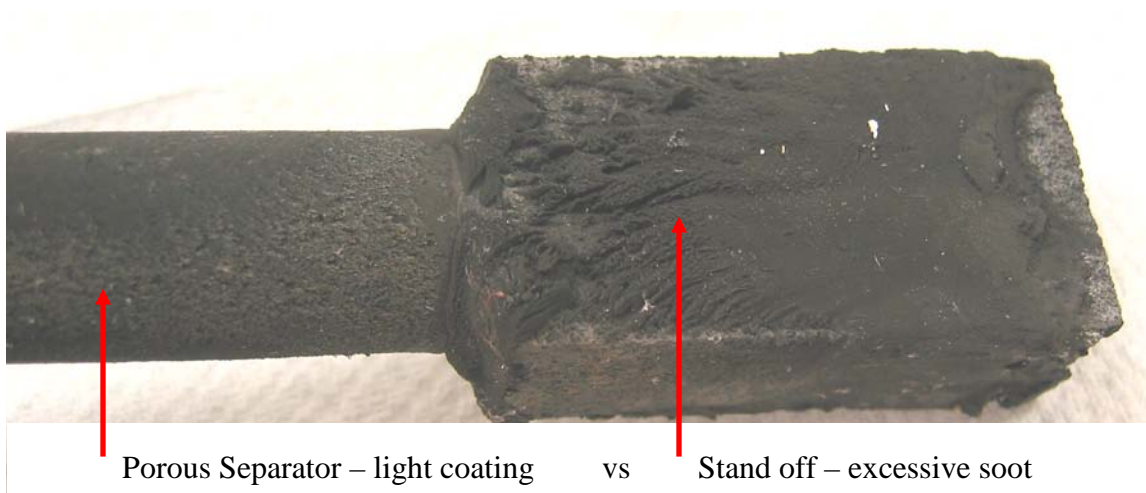


Figure 10: The porous separator and stand off illustrating a light coating of amorphous carbon on the separator and excessive soot build up on the stand off.

The JP-8 flow rate used during the testing single cell shown in Figure 10 was 10 $\mu\text{l}/\text{min}$. The excessive soot build up at the bottom is not unexpected. Antidotal evidence suggests that the thermal mass of the JP-8 droplets is sufficient that the droplet will reach the bottom of the anode chamber before complete thermal decomposition occurs. Therefore the soot formation tends to be heaviest at the bottom of the anode chamber. This concept was further supported when cells were thermally cycled at extremely high JP-8 flow rates. This led to the bottom half of the cell being covered with a dense layer of carbon during thermal cycling of the cell, Figure 7. However, the cell was still able to draw power, Figure 5.

The use of high JP-8 flow rate, i.e. 50 $\mu\text{l}.\text{min}^{-1}$, at single cell level is due to:

- Single cell setup that is known to have air leaks, therefore more fuel is needed to ensure a reducing atmosphere.
- The time frame of the program was limited; therefore a high flow rate was used to deliberately induce the worst case for carbon build up.

The anode chamber at system level will be very different than the single cell setup used in this program. Dispersion of the fuel should be easier due to the size scale upwards. Initial system testing by CellTech has shown that the anode chamber can be designed to evenly distribute the fuel. The fuel flow rate required can be estimated by assuming stoichiometric combustion of JP-8 as Cetane ($C_{16}H_{34}$) in air. Figure 11 illustrates that a 10 $\mu\text{l}/\text{min}$ JP-8 flow rate will provide a reducing atmosphere if the air leak rate into the anode chamber is less than 92 sccm. It is anticipated that the fuel flow rate can be further reduced in subsequent trials, since the exhaust is rich in black smoke. The presence of black smoke suggests a fuel rich reducing environment. A fuel flow rate of 5 $\mu\text{l}/\text{min}$ during cycling should be possible. Since stoichiometric combustion of 5 $\mu\text{l}/\text{min}$ would require 46 sccm of air.

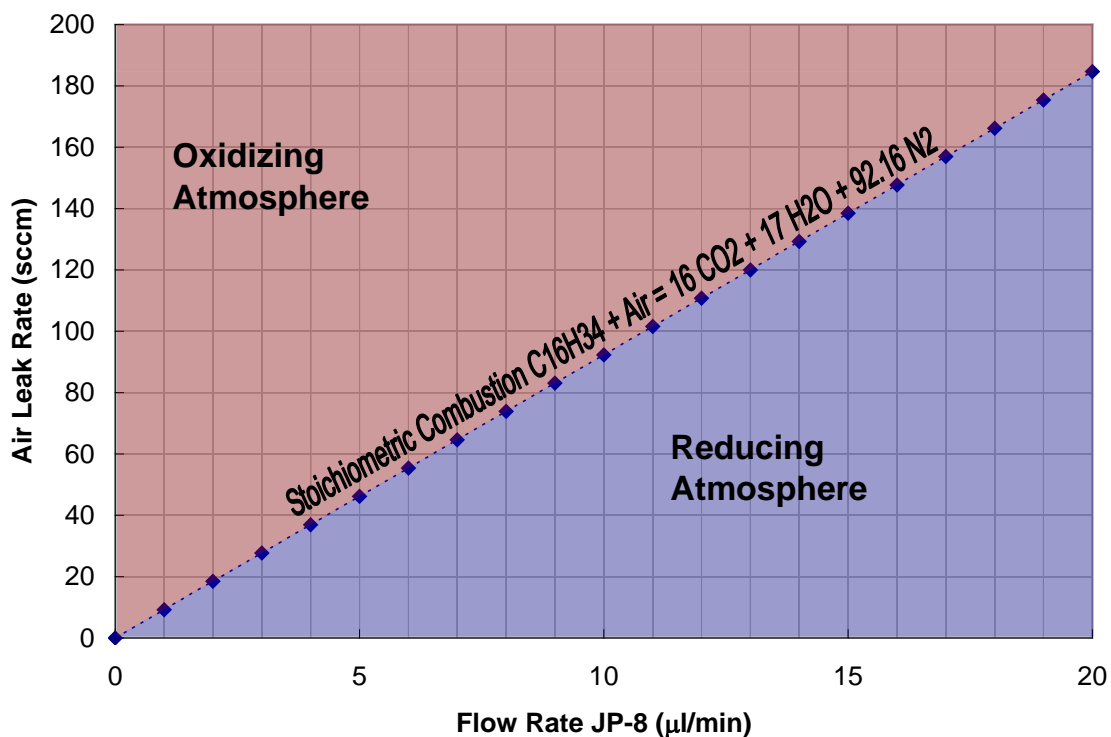


Figure 11: The JP-8 flow rate as a function of air leak rate

The anode chamber used in initial testing is stainless steel with an insulation layer on the inside. This chamber is currently sealed using compliant cold zone gaskets. As the design matures it is expected that the compliant seals will be replaced by a welded seams. This will greatly reduce any air leakages into the anode chamber. Thus the required fuel rate will drop substantially. It is anticipated that the flow rate would not need to be greater

then $5 \mu\text{l} \cdot \text{min}^{-1}$ for a 250 W system. It is also expected that the heating rate would be substantially greater. Current testing of stainless steel anode chamber has allowed 1000°C to be reached in fewer than 15 mins. The heating rate is not linear. The anode chamber very quickly reaches 400°C , ~ 5 mins. At this point the JP-8 will need to be introduced into the anode chamber to prevent oxidation of the tin and non-precious metal current collectors. Therefore, the JP-8 would be flowing for approximately 10 mins before the cells are operational and consuming the carbon deposits. Allowing for a similar time for cool down, a total amount of $100 \mu\text{l}$ would have been injected into the anode chamber. For a 250 W system the current projection is 64 cells. This means a $\sim 1.5 \mu\text{l}$ of JP-8 per cell. Since the system anode chamber will be design to even deposit the soot across all 64 cells, it is anticipated that each cell will only be lightly coated with soot.

The system controls will also allow for an automated tapering of the fuel flow rate based on temperature and Open Circuit Voltage (OCV) of the cells. During sub-stack testing the OCV as function of temperature for JP-8 can be empirically determined. A lower limit at which the OCV of the anode chamber's atmosphere is oxidizing enough to cause detrimental SnO_2 formation can be determined as a function of temperature. The fuel can then be injected intermittently when the OCV indicates the atmosphere is changing to an oxidizing environment.

Therefore, to summarized the solution for preventing excessive sooting of the porous separator during start up and cool down:

- Much lower flow rate of fuel per cell will be used since the anode chamber will be welded shut and therefore have less air leaks then the single cell test setup
- The heat-up and cool-down rates are much higher at the system level which reduces the time (and therefore reduced the total amount of fuel injected) before the cell reaches an operational status. Therefore soot build up will be limited.
- System control software can determine when the OCV of the cells indicate that the anode chamber environment is becoming oxidizing atmosphere and intermittently meter the fuel.

Metallic Interconnects: The non-precious metallic current collector used is proprietary. The natural metallic oxide layer that forms on the surface does not provide sufficient protection in an elevated temperature oxidizing environment. Therefore potential solutions are:

- component is coated with a protective layer, or
- component is submersed in a reducing atmosphere

Experimentation using a single cell demonstrated successfully the strategy of submersion of the component in a reducing atmosphere such as hydrogen or 95%/5% H_2 . In this program, one of the main goals is to demonstrate the component can be protected during start up, run and cool down with JP-8 only without the use of hydrogen or Ar/ H_2 . Inside

the anode chamber this is easily achieved by exposure of the metallic interconnect to the JP-8 environment. However, above the anode chamber the metallic interconnect is exposed to a hot oxidizing atmosphere. This meant that a small amount of reducing atmosphere had to be exhausted around the metallic interconnect that was above the anode chamber. Another concern is that soot build up inside the anode chamber could bridge from anode to the cathode current collector causing circuit shortage. These two concerns were solved synergistically using alumina weaved sleeve that was impregnated with ceramic glue, Figure 12. This sleeve was porous enough to maintain a reducing atmosphere around the metallic interconnect, however, the sleeve was also dense enough to provide a barrier to soot. A quantifiable result was obtained by a four probe resistance measurement of the metallic interconnect after testing, Figure 13.

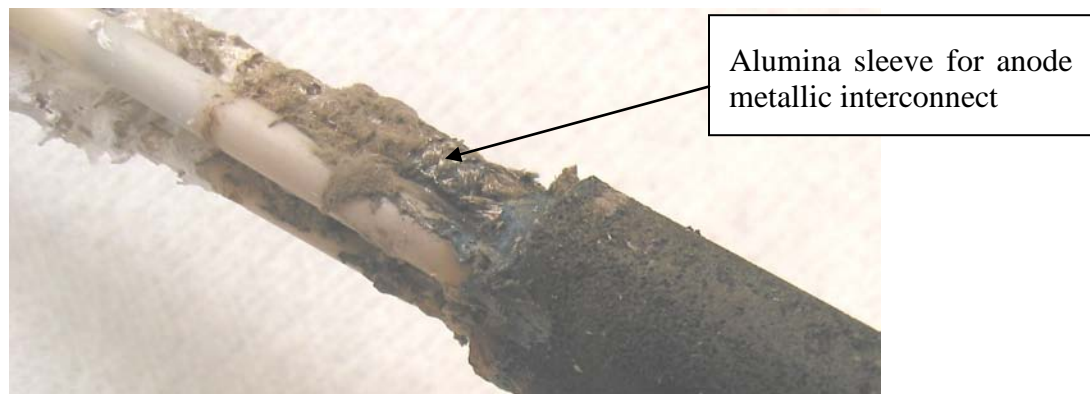


Figure 12: A single cell showing the alumina sleeve that protected the metallic interconnect from soot build up inside the anode chamber; and, also exhausted the anode gas around the metallic interconnect above the anode chamber.

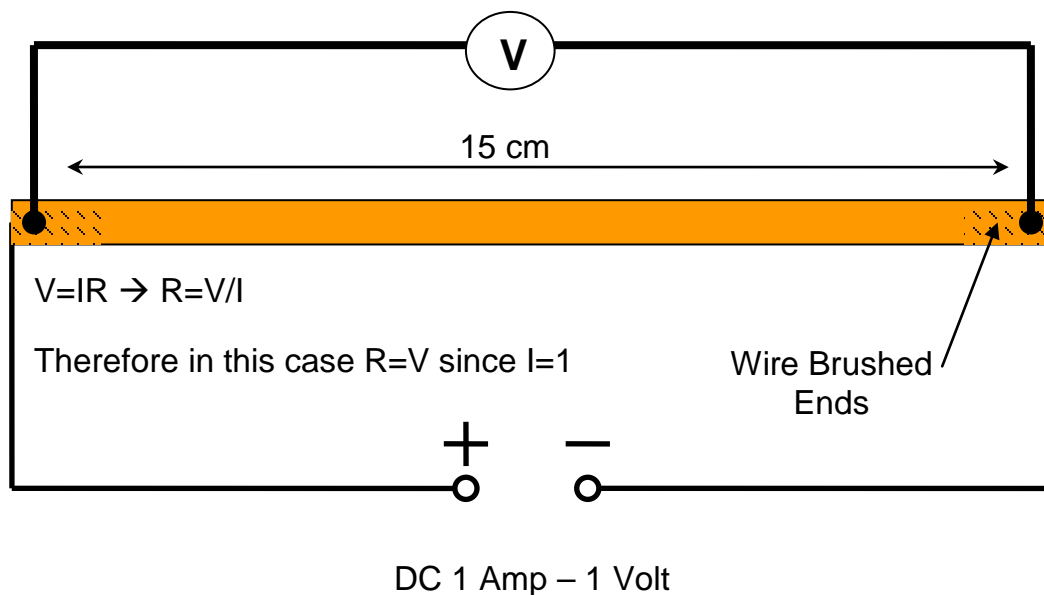


Figure 13: A schematic representation of the 4 probe resistance measurement

This result was compared to a new metallic interconnect and the 2nd metallic interconnect of the cell that was protected using a constant stream of 95% Ar / 5% H₂ gas, Table 1. These results illustrate that anode gas protected metallic interconnects would increase the resistance at operational temperature by less than 0.25 mΩ per cell compared to a new interconnect. It is expected that the increase in resistance of the metallic interconnect is mainly a function of thermal cycling, and not highly dependent on time at operation temperature. Only the length of metallic interconnects inside the anode chamber was used for the calculation. Stacking of single cells in a system will making the length of metallic interconnect outside the anode chamber redundant. The increase in resistance compared to a new metallic interconnect was due to a small amount of surface oxidation. One concern is that subsequent thermal cycles may increase the amount of oxidation. The significance of thermal cycling on metallic interconnect resistance is still to be evaluated.

Table 1: 4 Point Probe Resistance Measurements

Measured	New	95/5 Gas Protected	Anode Gas Protected
Resistance ($\mu\Omega\cdot\text{cm}^{-1}$)	50	52	55
Δ Resistance ($\mu\Omega\cdot\text{cm}^{-1}$)		2	5
Calculated			
Resistance @ 1000°C ($\mu\Omega\cdot\text{cm}^{-1}$)	385	397	417
Δ Resistance @ 1000°C ($\mu\Omega\cdot\text{cm}^{-1}$)		12	21
Resistance @ 1000°C (mΩ/cell)	3.85	3.97	4.17
Δ Resistance @ 1000°C ($\mu\Omega/\text{cell}$)		115	205

At this stage no experimental research has been performed on coating of the metallic interconnect. Commercial technology is readily available and suitable for electroplating of the metallic interconnects. Handbooks on coatings suggest that a secondary metallic coating can applied that will form a very hard impenetrable oxide layer on the surface. Although coating of the metallic interconnects are theoretically not needed when the metallic interconnects are submersed in a reducing atmosphere, they would provide redundancy to prevent oxidation.

The protection of the metallic interconnects can be summarized as:

- Submersion of the interconnects in the reducing atmosphere of the anode chamber.
- Isolation of the metallic interconnects from each other using a loosely weaved alumina sleeve.
- Coating of the metallic interconnects with an inert oxide layer as a redundancy measure.

Tin penetration of the porous separator: The retention of the tin inside the porous separator using only the surface tension has been proven to work for the current Gen3.1 cell design during single cell testing using JP-8 as fuel. However, during start up and cool down previous tests had used hydrogen. In this program only JP-8 was to be used. One of the main goals of this program is to demonstrate porous separator's capability to contain tin even at excess sooting/carbon formation during startup and cool down. As can be seen in Figure 8 and Figure 9 there was no movement or leakage of the tin through the porous separator even under soot forming conditions. It is suggested that any discreet amounts of tin observed in the micrographs are due to pathways greater than 300 μm near the inner diameter of the porous separator. Under other ongoing programs there is substantial effort directed towards improvement of the porous separator. It is anticipated that a change in manufacturing method will ensure greater homogeneity of the porosity and eliminate damage to the inner bore from the current final machining operation.

Fuel Inlet and Exhaust Port on Anode Chamber: In previous programs clogging of the fuel inlet and exhaust ports on the anode chamber have been a sporadic problem. Alignment of the needle that meters the fuel into the chamber has helped to eliminate this problem. It is possible that partial thermal decomposition of the fuel droplet causes a carbon rich vapor in the inlet tube. The carbon rich vapor appears to provide an ideal environment for the growth of equal axial carbon whiskers on the inside circumference of the alumina tube, Figure 14.



Figure 14: Carbon whisker growth observed on a single cell fuel inlet port from a previous program.

It is suggested that changing the nucleation sites for whisker growth may solve the issue. The alumina surface that forms the inside the anode chamber grows a very thin graphitic carbon layer. Apart from dimensional difference in the alumina surfaces, the anode chamber is formed by slip casting while the fuel inlet and exhaust tubes are extruded. It is reasonable to assume that the surface finish is significantly different between these different manufacturing methods. It is also apparent that the height of the inlet and

exhaust influences the soot formation. Therefore the strategy for eliminating clogging will be as follows:

- Ensure correct alignment of the fuel injection needle
- Vary the height of the fuel inlet and exhaust tubes
- experimentation with coating on the inside of the tubes to change the nucleation properties of the surface.
- Limit fuel flow rate during heat up and cooling to avoid excess soot build up.

To date these strategies have been implemented into the system design process. Testing of the stack box design with single cells at the end of 2008 will verify these design concepts.

Conclusions

This program was to demonstrate the feasibility to heat-up and cool-down of CellTech Power's Gen3.1 cells on JP-8 fuel only without use of additional reducing gases such as hydrogen or Ar/H₂. The program was able to successfully demonstrate the following.

- A successful operation of single cell using JP-8 only as a protection media during a complete cycle: startup, run and cool down.
- High levels of sooting/carbon deposition on the porous separator due to excess fueling did not prevent the cells from functioning, although there was a decrease in cell performance.
- Thermal cycling the Gen3.1 cell using JP-8 only even at excess sooting/carbon formation did not result in additional tin penetration through the porous separator.
- The non-precious metal current collectors were successfully protected using JP-8 only or the anode exhaust gas as a reducing atmosphere without significant loss of performance.

This program, therefore, achieved its goals to demonstrate feasibility of JP-8 only for startup and cool down

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